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**STAGED EXPLOSIVE TO MITIGATE COOK-OFF VIOLENCE:
PRELIMINARY WORK WITH 2,4-DINITROPHENYL HYDRAZINE**

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14. ABSTRACT A potential method to mitigate cook-off violence of heavily confined explosives is to add a minor ingredient that reacts at temperatures below where cook off usually occurs, to burst the confinement and/or interfere with the thermal decomposition chemistry of the explosive so to slow subsequent reactions. This additive would not impact the explosive's performance significantly, but only comes into play during relatively slow thermal heating. The material 2,4-dinitrophenyl hydrazine has shown promise to produce the required mitigation for Composition B explosive in a laboratory confinement that mimics very heavy confinement. The results of this work are presented.					
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INTRODUCTION

We have been trying to use an energetic additive in a melt cast explosive that reacts at slightly lower temperature than the main explosive ingredients, to burst open any confinement before the main constituents react violently. Our early focus was to mitigate the cook-off violence of Comp B explosive. Through trial and error, some compounds were identified that maintained a separate exotherm from Comp B in differential scanning calorimetry (DSC) and larger scale DSC type tests at about the proper temperature. These were tried in a laboratory slow cook-off fixture holding about 80 g of explosive mixture that was heated at 6°F/hr until reaction [the U.S. Army Armament Research, Development and Engineering Center (ARDEC), Picatinny, New Jersey Scaled Thermal Explosion Experiment [STEX] test]. The fixture was capable of holding 27,600 psi hydrostatically, but was equipped with a small vent hole for these tests except for one trial. The vent was intended to make the bursting more difficult and is expected to mimic leaky, real world confinements. The initial work towards this goal was with adding pentacrythrite tetranitrate (PETN) to TNT (ref. 1). Results were encouraging. Next, the effort was shifted to mitigating Comp B. Pentacrythrite tetra-nitrate was removed from consideration, and a wide-ranging search for other additives was initiated. The criteria were that they react at lower temperature than RDX, but not too low, and demonstrate separate exotherms in DSC and larger, gram size DSC type tests. One energetic additive found by ATK Thiokol Propulsion (ref. 2), under contract, did significantly reduce violence in some initial trials and was subjected to further study in our facility. This was 2,4-Dinitrophenylhydrazine (DNPH).

Table 1
DNPH

CAS registry number	119-26-6
Density	1.659 g/cm ³ (S.T.P. RDX 1.902 g/cm ³ , melted (176°F TNT 1.472 g/cm ³)
Melting temperature	381°F (TNT 176°F, RDX ~ 383°F)

Initial safety testing of 4% DNPH with Comp B indicated: ABL impact = 13 (neat Comp B = 21), ABL friction ~ Comp B, ESD ~ Comp B, and vacuum stability 2.221 mL/g. The test material was 4% DNPH dissolved in molten (200°F) Comp B and panned out. This vacuum stability result was of concern. The second set of safety tests on the formulation, done at ARDEC, showed ERL impact 50% point = 54.8 +/- 1.2 cm (Comp B = 35.9 +/- 0.33 cm.), ESD (Picatinny Arsenal method) no reactions in 20 trials at 0.25 J, and ABL friction test, no reactions in 20 trials at 1800 psi. Note: impact sensitivity reversed itself. Vacuum stability showed 6.59 mL gas from 5 g mixture (Comp B gave 0.62 mL for 5.6 g). This was better, at 1.32 mL/g, than in the first set of tests, and passes the requirement in MIL STD 1701B for compatibility. However, the cause of the gases still needed to be determined. Further tests used a 50/50 DNPH/Comp B formulation, intended to amplify any interaction. However, the DNPH was dried at 230°F for 8 hrs and the Comp B was dried at 158°F for 8 hrs before melting the Comp B and adding DNPH. Then the 50/50 formulation mixture was heat conditioned for 216 hrs at up to 212°F (above melting). Neither DSC nor Fourier transform infrared (FTIR) analysis, done at intermediate times and at the final time, indicated any change from the virgin materials. Evidence was strong that they are, in fact, compatible. It is speculated that the gases seen in the original formulation may have been moisture.

Another explosive of interest is PAX/AFX 194. In contrast to Comp B, this explosive just has RDX in an inert binder. There is no TNT in this formulation. The RDX used is somewhat different from that in Comp B, in that it has no HMX impurities. This causes the DSC to display a much cleaner melting endotherm than when HMX is present. But, the main exotherm appears the same between the two types of RDXs. The exotherm is also the same between the two RDXs when they are incorporated in the PAX formulation.

DIFFERENTIAL SCANNING CALORIMETRY

Most DSCs were done with 50/50 physical mixes of DNPH and explosive. Figure 1 shows small interaction between DNPH (Candidate 2) and RDX. This was standard United States RDX. It appears the reaction of DNPH was shifted to lower temperatures when mixed with RDX. Note: exotherms of DNPH and RDX stay distinct. The DSCs of DNPH and TNT (fig. 2) indicate a major change. It seems disturbances where neat DNPH should react are minimized, while the exotherm that apparently corresponds to TNT, shifts to lower a temperature by a large amount. But, this temperature is still significantly higher than that where neat RDX has its exotherm. One would expect that RDX would still dominate cook off in a formulation having both TNT and RDX. Figure 3 shows DSC of Comp B with 4% DNPH in the formulation. Certain temperatures are marked for reference later in the discussion. In contrast to these results, DSC of DNPH and PAX failed to show any noticeable interaction at all. The 7% DNPH/93% PAX formulation looks like the neat DNPH and RDX material plots superposed on one another.

TESTING PROGRAM

A number of STEX tests were completed with DNPH formulated with Comp B and a single test using DNPH with PAX/AFX 194. Inconsistent results were obtained. Sometimes the violence level was surprisingly mild, although sometimes it looks as violent as neat Comp B. We are trying to understand how the mild results occur and see if this can lead to the desired mitigation of cook-off violence.

The STEX test is a 1-in. diameter cylinder, 4 in. long, sealed at each end with massive end plates. This size is similar to the sample size used in the variable confinement cook-off test [sic] [VCCT (ref. 1)]. Down the axis is a probe containing five uniformly spaced thermocouples, with the end thermocouples only 0.100 in. from the end plates. There are two thermocouples on the outside, covered by foil, with band heaters over wrapping the assembly. Larger band heaters over wrap the massive end plates. Heaters are controlled by the two external thermocouples. One centered thermocouple controls the central band heaters and the other, end located thermocouple, controls the heaters on the end plates. It is heated at 6.6°F/hr. The hydrostatic strength (bursting pressure) of this cylinder is similar to that of a typical heavy bursting projectile, which is about 30,000 psi. Results observed for each test are given in the spreadsheet (fig. 4).

Neat Comp B and the DNPH/PAX mixture do not have a low temperature exotherm at 260°F in the STEX test. None of the DSCs indicate any exotherm at this temperature, even the DNPH/Comp B mixture (260°F is indicated by the "126°C" mark in figure 3). However, all STEX tests with DNPH/Comp B have this exotherm and its magnitude depends on the amount of

DNPH. This exotherm occurs only when the mixture has both DNPH and TNT. There is no observable action associated with this exotherm. That is, the usual slow bubbling of liquid from the vent hole that occurs throughout a test does not change character as a result of this exotherm. Whatever it is, it does not seem to produce a noticeable increase in gas output. It is unknown why the exotherm is not observed in DSC results.

DISCUSSION

The results of using DNPH with Comp B are summarized in table 2. Shot to shot variations stand out. As examples of the difference between mild and violent reactions, figures 5 and 6 show two mild results that contrast with violent neat Comp B in figure 7. The object of the trial without vent was to verify that venting is a penalty in this test. Other authors (ref. 4) have noted that leaking led to greater violence. The no vent trial did not produce any fragments at all, but could be considered to be only incrementally better than other mild results, perhaps a random variation. In any event, plugging the vent did not lead to increased violence.

Table 2
Results of STEX tests

Comp B		PAX/AFX194	
Number of replications	Violence	Number of replications	Violence
3	Violent (= Comp B)	1	Violent
2	Reduced		
3	Mild		
1	No explosion (ejected through vent)		

There is no absolute correlation of violence with the percent of DNPH, cook-off temperature (highest temperature recorded on outside of the fixture), highest internal temperature, or position within the fixture where runaway started (taken as the position of the thermocouple that appears to start the ramp to explosion). The debris from a DNPH/Comp B mild reaction more-or-less resembles debris from neat TNT (fig. 7) in the same test, even including the massive black deposits left on fixture parts. But, deposits from DNPH/Comp B tests seem to have a more leathery quality (sort of a thick film coating) than the big, chunky, dry soot like deposits from neat TNT. The two neat TNT trials gave identical debris. The internal temperature recording system was not so good when TNT was tested; however, the external cook-off temperatures were 470°F and 467°F. This is much hotter than any temperature observed when RDX is in the system, as expected. The almost TNT like nature to the mild reactions at relatively low temperatures is a surprise. Unfortunately, results were variable. Figure 8 is also a DNPH/Comp B test, and it is hard to see any difference from neat Comp B.

All the mild reactions left a black to brownish, tar-like residue on the pieces of the fixture. Objects adjacent to the test were sprayed with tar-like deposits. For example, once a nearby (8 in. away) light bulb survived a mild reaction (fig. 4), but had the side facing the fixture coated with a spray of "dried tar." None of the reduced violence results had this type of deposit. They had more of gray soot. The violent reactions left only minimal deposits of dark gray soot. Neat Comp B and the PAX mixture were in this category. We were not equipped to recover explosive

in the blast chamber. However, one test left several big hunks, later burned in place. Generally, we didn't readily find such pieces, and any small crumbs were destroyed during decontamination of the chamber.

The five equally spaced thermocouples are top, upper, center, lower, and bottom. Thermal runaway, as detected by internal thermocouples, typically started in the lower half of the fixture, most often at the lower thermocouple (not the bottom one). But, once it started at the bottom, once at the center, and once at the top thermocouple. There is no obvious relation between where runaway starts and violence level. Since these tests heat up very slowly, there is time for RDX and DNPH to settle from the less dense melted TNT. Sometimes there is evidence of convection inside the fixture as the cook off temperature is approached. That is, a temperature rise at a lower thermocouple is seen, after delay, at the next higher thermocouple, and so on. Always, there are both exotherms and endotherms (temperature change 10 ~ 30°F) during the period just prior to explosion. Often, cooling is seen on several thermocouples during this time. Mostly, the bottom thermocouple rises monotonically, with, at most, a plateau when others are cooling.

Fast convective movements probably are not distinguishable from the noise in the recording system or are hidden between data points. Once we were able to correlate strong gas venting with thermocouple readings. Unfortunately, this particular test was special, because it had some kind of programming failure for the end plate heaters, causing nonuniform conditions. The top and bottom thermocouples read 307°F, the center and upper thermocouples read 340°F, while the lower thermocouple read 360°F. Then an event occurred where the top thermocouple had a sudden jump to 330°F as the bottom and lower thermocouples dropped from their respective temperatures by about the same amount. Most thermocouples had their jump between data points (less than 0.17 sec.), but the center and upper ones only showed temperature drifting down over 1~1.5 min. During these 1~1.5 min, the top station gradually cooled and the bottom and lower stations gradually heated, bringing the bottom up to the temperature of the others. Accompanying the sudden jump, video shows two strong "puffs" of reddish gas, each bright enough to saturate the video for one frame before dieing out. Each puff event only occupied 3~4 video frames (about 0.8 ms) and there was only one frame between them. Observations returned too normal during the 1~1.5 min of gradual adjustments. There was not much bubbling for a long time before the puffs. The loss of bubbling corresponded to the loss of programmed heat on the end plates that occurred some 550 min before the puffs. Usual bubbling started up again after the puffs and continued until explosion some 63 min later. The temperature of this jetting corresponds to the range 329 ~ 360°F (indicated as the 165 - 182°C region in figure 3). There is a range because not all internal thermocouples report the same temperature and the location of reaction within the fixture cannot be positively identified. The range is the variation between thermocouples at the moment when jetting is observed. There is an exotherm on the DSC of figure 3 in that range. No jetting was seen on other videos, but not every trial had video. One non-video trial had almost identical thermocouple signal at about the same temperature. Very likely this one had jetting also. Its violence was also mild, just like the videoed one. It did not suffer end plate heater malfunction. The very strong endothermic effect with this jetting suggests that the weaker endotherms seen near explosion could be from mild gas generation. Although bubbling of liquid continues through a test, sometimes a crust forms on top of the fixture over the vent hole. Occasionally this crust blows away, and afterwards just slow bubbling continues. This could be an occasional surge of gas release associated with small endotherms inside the fixture. Test 8, without vent,

had the smoothest internal data of any test. All stations smoothly ramped up without any cross-overs of the data or temperature decreases prior to explosion. The station with highest temperature throughout this test (the lower station) led thermal runaway. Temperature at which runaway occurred were equivalent to where the thermocouple signals show gyrations in vented trials. There was nothing special about the general magnitudes of internal temperatures associated with this particular cook off. But, the external temperature was noticeably lower than nominal. We concluded, the normal heating and cooling gyrations which start about 30 min or so prior to explosion, even with neat Comp B, probably are caused by some combination of convection inside the fixture and spurts of gas venting.

Violence might relate to aging. The 5% sample tested within 2~3 weeks of casting gave no explosion, while its twin tested more than 3 months later was violent. The 7% sample tested within 2~3 weeks of casting gave mild results, while its twin tested 7 months later was violent. Another 7% sample made and tested within 1 month was mild (but this one was also different in that it did not have any vent). Not much study specifically related to aging was accomplished.

CONCLUSION

Using the heating rate employed here, Wardell and Maienschein (ref. 4) report Comp B giving a mild response in 14508 psi confinement and violent response in 29016 psi confinement with their 2-in. version of the Scaled Thermal Explosion Experiment (STEX) test (the original version of the test). They noted violence was a function of heating rate. When they used 1.8°F/hr, detonative reaction occurred once in one trial of the lighter confinement and of two trials in the heavy confinement, one was detonative and one violent. A heavy confinement trial at 3.6°F/hr was violent. All these results were obtained with tightly sealed test fixtures. For reference, in the heavy confinement fixture at 1.8°F/hr, PBXN-109 gave a mild reaction (only three large, slow pieces of the confinement were ejected) in one trial.

In the variable confinement cook-off test [sic] (VCCT) test, Comp B gave one mild and one violent reaction at a rated confinement pressure of only 2350 psi and only violent reactions at higher confinements. The VCCT depends on compression of the confinement to make a seal at the end plates. As the explosive melts, it expands the confinement, which has the effect of removing the compression seal of the end plates. Leaking ensues, characterized by melted explosive running out of the fixture over a long period of time prior to reaction. Reaction gases also follow this leak path. So, these tests are sort of a vented situation, with the amount of venting uncontrolled. This test was heated at 6.6°F/hr.

The U.S. Army Armament Research, Development and Engineering Center (ARDEC) STEX test does not exactly duplicate either of these. We use the higher heating rate and heavy confinement that gave the violent result in reference 1, but the ARDEC test is half scale. We have the same scale as VCCT and venting too, but our confinement is much heavier. So, by comparison, the mild reactions that were obtained with 2,4-Dinitrophenylhydrazine additive show considerable improvement in cook-off violence, and may offer a means to mitigate cook-off violence in heavy walled bursting projectiles. But, the reason for variability in the results must be understood and eliminated first.

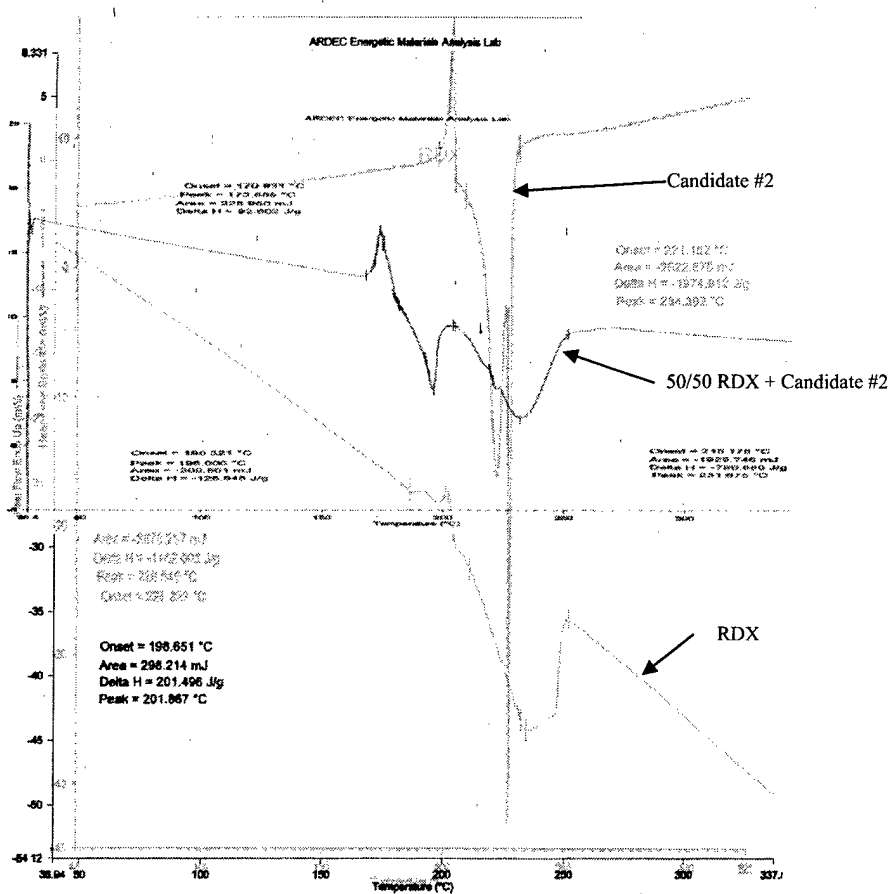


Figure 1
DSC's of DNPH with RDX

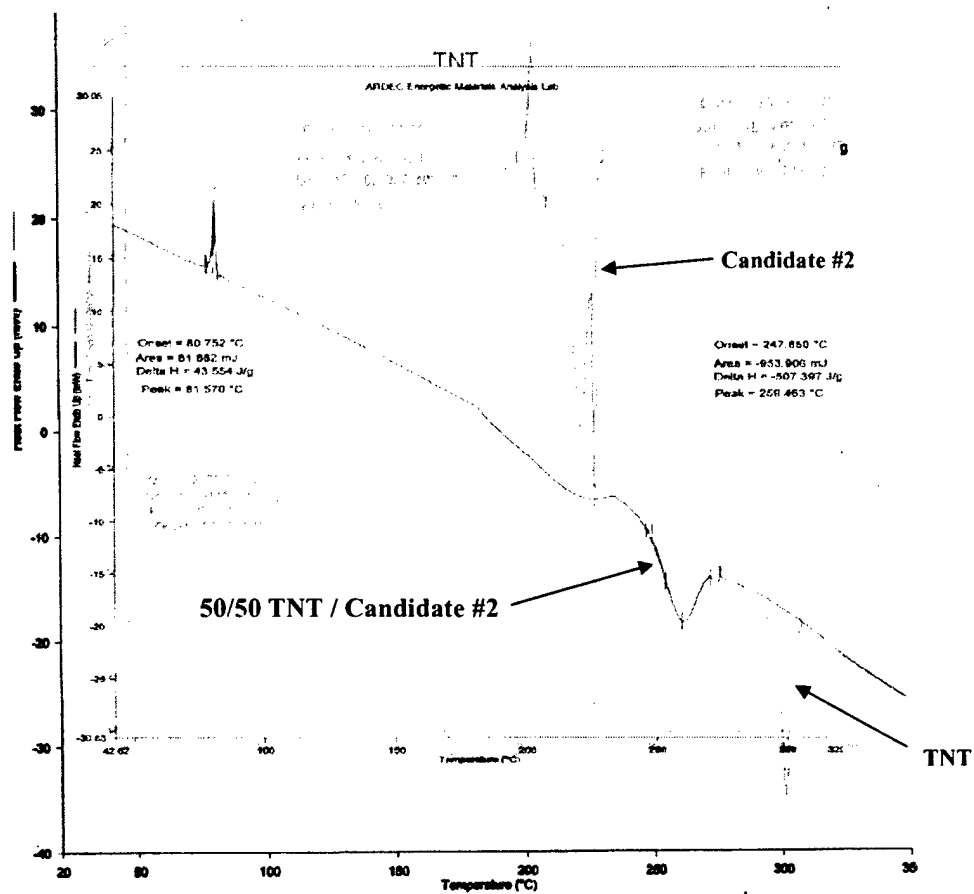


Figure 2
DSCs of DNPH with TNT

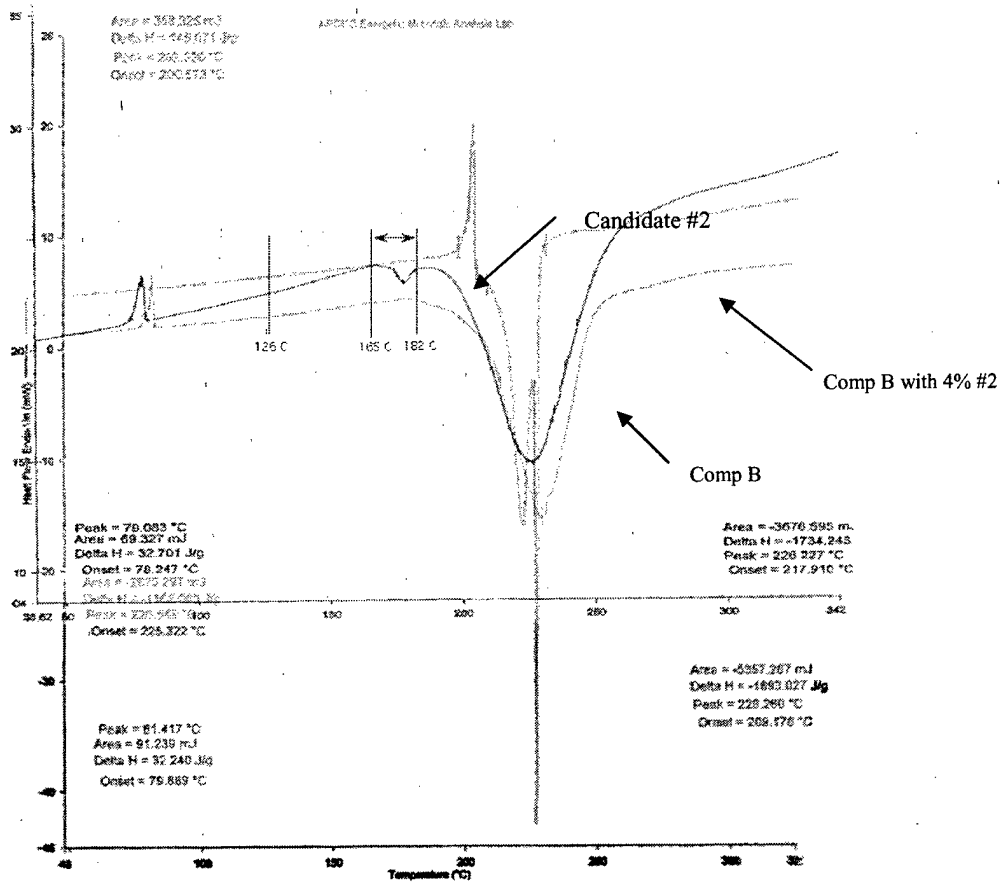


Figure 3
DSCs of DNPH with Comp B

Test ID	exotherm 1 260 - 270 range temperature in Fahrenheit		endotherm	time interval until next event	exotherm 2 310 - 330 range		endotherm	time interval until next event	exotherm 3 340 - 360 range		runaway to explosion	explosion	accuracy comment	TNT by volume				
	start	peak			start	peak			start	peak				T1	T2	T3	T4	T5
TEST 1: Comp B 12/5/2001	None		None		319-332 Simultaneous all stations.	T1 & T2 ramp to 364-369 over 110m. T3 & T4 similar with 20m delay in peak. T5 plateaus at 346 for 15m.	T1 & T2 cool over 35m. T3 & T4 cool over 10m. Finally T1-T2-T3-(360) with T4 about 6 degrees higher (~366).	0	T5 ramps up from its 346 plateau with T4 following after 8m. delay.	None	T1 & T2 & T3 keep cooling until just before explosion. (max 363). Only T4 cools. T4 only cools to 368 before starting up with same slope as T5.	VIOLENT But not detonation.	T1 - T5 spread across 7°F band with T5 lowest and T2 highest. Runaway starts at T5 even though it is cooler. Max=348	Blotchy, dk. gray flaky deposits on top cover - bottom fairly clean.				
TEST 2: DNPH/Comp B 12-7-01	260-270 T3 slightly delayed	278 Duration = 88m. Very weak exotherm.	297-303 separate endotherm drops 1 after about 246m. Duration=15 m.	200m. (from separate endotherm. not from exotherm #1)	312-333 all stations ramp over 138m.	T1=T2=361 T3=T4=351. T5 has no peak. All except T5 peak simultaneously	All drop 8-10 over 10m. except T5 continues up.	30m. but internal heating is obvious.	T1&T2&T4 stayed steady -355 over 45m. in spite of external ramp. T5 continues exotherm #2.	No Peak. T3 jolts ramp up of T5. Eventually these meet others so all stations report ~ 355.22m. prior to explosion.	REDUCED	T1 & T2 high by 4-5°F. T5 low by 4°F. About 8°F spread. Max = 349	Slight dry grey soot coat on top - cleaner than Comp B.					
TEST 3: 1-9-02	265 lags behind, has smaller signal.	T5 275 Duration = 90m.	None	435m.	322 Gradual ramp.	T2=357, T5=348 Duration = 230m.	Drop 20 in less than 1m (between data points). Except T1 drops 10 over 10m. Top & Bottom external stations have disturbance associated with this event. But center external shows steady heating as intended. Possible data problem?	0	T1=348. T5=338 but with much steeper slope it eventually becomes hotter than others.	T5 ramps to 362 over 55m. T5 ramps up to meet T4, then they track over 30m. Others fall behind.	MILD	T2 high by 1.4°F T5 low by 4°F	Significant tar-like black deposit on all surfaces					
TEST 4: 6-7-02 apparently cast in May VIDEO											REDUCED			Very light gray dry soot in some places - cleaner than Comp B				

Figure 4
STEX results using DNPH with comp B

TEST 5: 5% 282-288 8-2-02 apparently cast in July (exact date uncertain)	278 Duration = 100m.	None	502m.	332 Except T4 & T5=386 T1=381 T3 were not affected very much.	T5 & T1 & T4 drop 30 over less than 0.17m. T4 & T3 not affected.	354, but T5 lags behind. Other stations track.	388, but T5 lags behind. Duration = 150m.	None	All except T5 show exponential ramp with "knee" at 389. Then go to 355-600 in less than 0.17m. T5 external heating then continued up to 376 and suddenly jumps to high value with other stations. Max various=398	NONE Runaway did not damage the fixture or probe. All stations cooled to 355-388 within 1m. Since external heating up to 410-415, the internal probe followed after recovering from the initial cooling. No further events occurred.	T4 high by 4°F T5 low by 3°F	External temperature reached 336°F at final runaway. All stations except T5 reported simultaneously. Significant material deposits. Deposits on ground underneath. Much from the weep hole on top. It is possible leakage also occurred out the bottom - but it was not obvious any did. Max=338 then system kept heating to 410.	Black, crusty deposits on outside from vent and over drip path to down, underneath fixture. Resembles usual deposits which accumulate around vent hole, but a more deposits. Deposits on ground underneath. Much from the weep hole on top. It is possible leakage also occurred out the bottom - but it was not obvious any did. Max=338 then system kept heating to 410.
TEST 6: 5% 11-22-02 cast in July with the sample tested 8-2-02 VIDEO	278 Duration = 50m.	separate endotherm at 278 some 50m. later	553m.	T5=332, T2=337	None. Very gradual ramp	T5=346	358 - 358 except T5 & T4 do not peak.	Only T1 & T2 & T3 cool 2-3 over 30m. while T5 & T4 continue to 379 sharply to 398. Max T4=398	T5 & T4 have ramped to 379 over 90m. Then T4 ramps sharply to 398. Max T4=398	VIOLENT	Good tracking, all within a band of 3°F	External temperature reached 347°F. Runaway seen at T4 & T5 simultaneously. Max = 347	Fairly clean - no significant deposits.
TEST 7: 7% 8-2-02 cast with the 5% samples in July	269 - 276	None	645m.	315 - 317 except T1 & T5 which are cold due to end heater failure.	T2 & T3 & T4 = 334	T4=332, T5=281, T1 & T2 & T3 do not show this exotherm. T5 is cool due to end heat failure.	T4 ramps to 360 over 60m. and then stays steady for 55m. T5 gradually reaches 310.	T1 suddenly jumps up by 17 to 333 in less than 0.17m. while all other stations except T5 jump down to this value. T5 jumps down 20 to 279, then rises to join the others 2m. Later. Couple of strong spurts of orange gas observed with the temperature drop..	T4 jumps up to 347 and stays roughly steady for 100m. while other stations are steady at 14 - 16 cooler. Max T4=349	MILD At T4=349 Lightbulb did not break.	T3 & T5 low by 6-7°F. End cover heaters had a partial failure -> sets up temperature gradient with T1 & T5 tracking 30 lower than T2 & T3 & T4. But this difference goes away suddenly about 100m. before exposure. Must indicate vigorous convection occurs inside fixture.	Extensive black, crusty deposits. Tar-like coating on nearby lightbulb.	
TEST 8: 7% 11-22-02 cast 10-25-02 no vent	-278	None	480m.	Not distinct. T3=303, others do not show exotherm.			T3 plateaus at 356 after 80m. T4 kind of plateaus at 361.	All stations drop 3-5 about 16m. before explosion, then ramp up. T4 rises more quickly.	T4 ramps to 388 (could be 377) in next 10m. and plateaus for 5m. T3 = 365. Max T4=388	MILD No fragments.	T4 high by 10°F others tracked okay. THIS ONE HAD NO VENT1	External temperature went to 325°F. This was a low cookoff temperature. Max = 325	Extensive black, crusty & tar-like deposits.

Figure 4
(continued)

TEST 9: 7% 252 2-21-03 cast with the 8 2-02 sample in July VIDEO	274 duration - 116m.	None	521min.	Not Distinct. T1 & T5 start monotonic ramp up at -320. T2 & T3 & T4 stay at slow heating rate.		zero	T1 & T5 plateau at 352 while T2, T3 & T4 don't show much heating right up until explosion.	ramp reaches T1 = T5 = 357 over 80 m. Finally T1 rises sharply above T5 in last 3 m. to 395. Next highest was T5=363. Max T1=395.	VIOLENT T4 was high by 10°F again.	External temperature went to 335°F. Appears T1 went up first - this never happened before. Also it is odd that T1 & T5 track each other. Max = 335	Thin, dry, dk. Gray soot coating.
TEST 10: 2 7% cast 21-03 2-14-03 explosive formation was MNX184 NOT COMP B	None			T1 T2 & T3 have an exotherm at 330. T4 & T5 only plateau. This exotherm is isolated - stations return to external ramp afterwards. Maybe because of IRDX.		All stations just return to follow externally imposed ramp.	T5 & T4 smoothly ramp up. T1 & T2 smoothly cool by 10 to 355 and plateau. T3 splits the difference, plateaus at 370 for 20m.	T5 = T4 = 387. T3 jumps sharply up to join T4 & T5 10m. before explosion.	VIOLENT T3 & T4 were 2°F high and T4 was 6°F high.	External temperature went to 353°F - a very high value. T4 seems to lead the way. Max = 353	Thin, dry, dk. Gray soot on some pieces.

Figure 4
(continued)

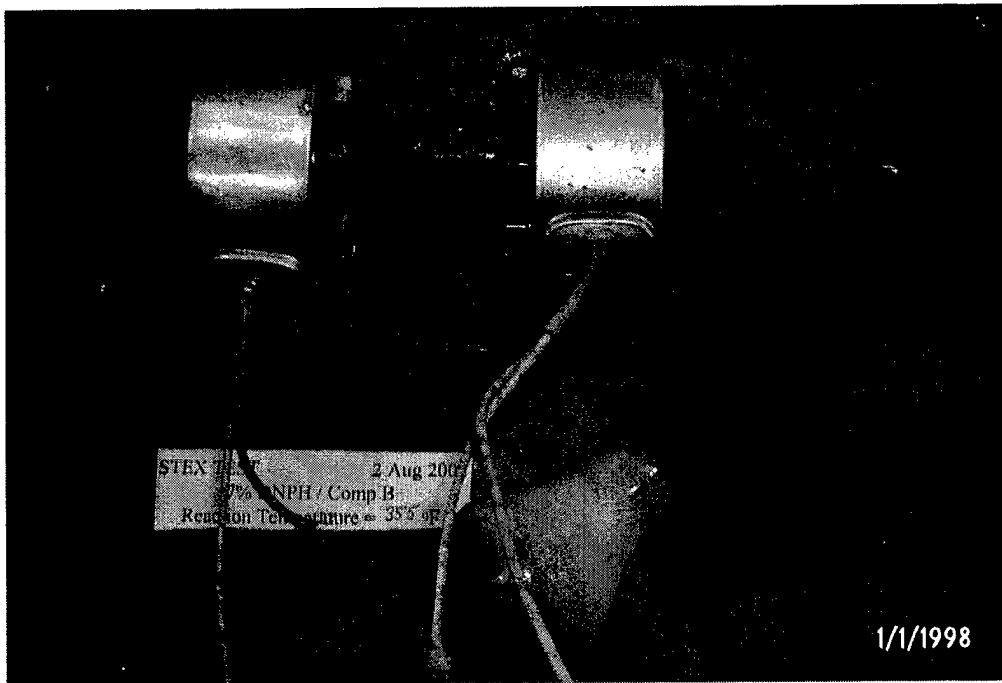


Figure 5
STEX test mild result using 7% DNPH with Comp B

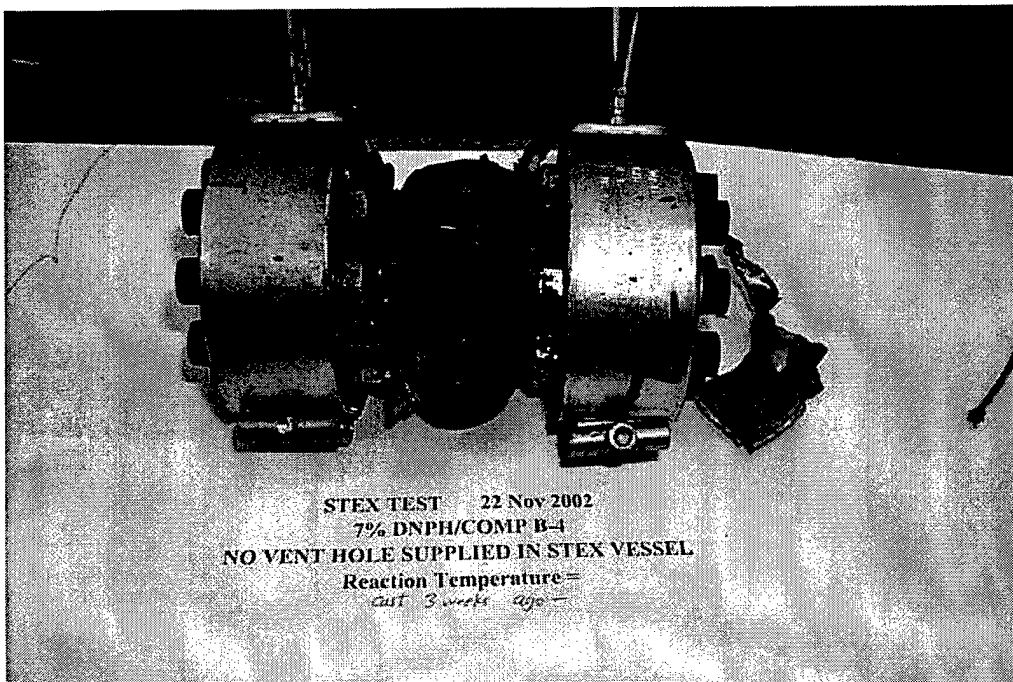


Figure 6
STEX test mild result using 7% DNPH with Comp B and no venting

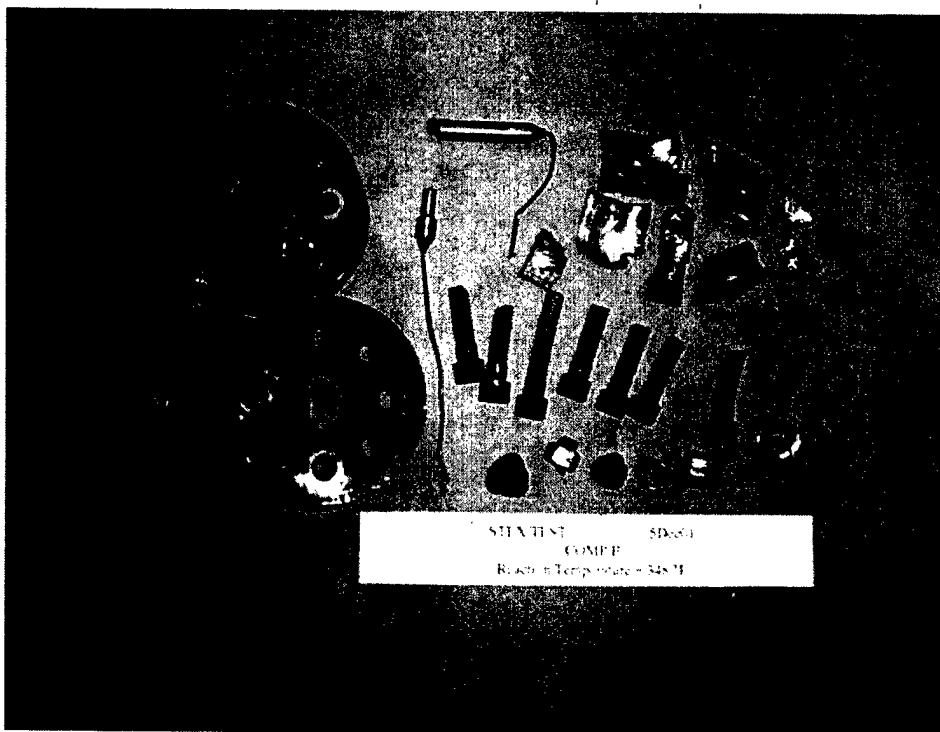


Figure 7
STEX test violent result using Comp B



Figure 8
STEX test mild result using TNT

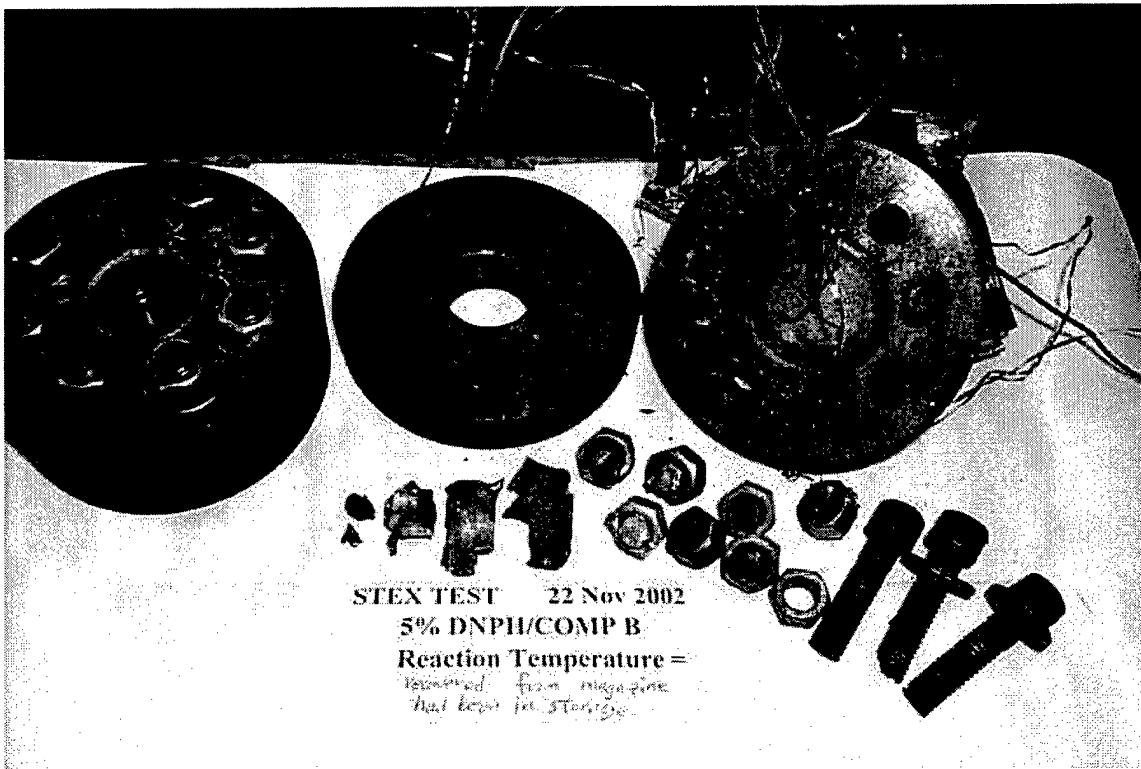


Figure 9
STEX test violent result using 5% DNPH with Comp B

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